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August 23, 1983

Task 23

Technical Support to Region V Re Asbestos Waste Site  
(Johns-Manville Corp.), Waukegan, IL

by

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## EXECUTIVE SUMMARY

The Johns-Manville Corporation operates a landfill disposal facility for asbestos waste material adjacent to its manufacturing plant at Waukegan, Illinois. As part of the evaluation of this asbestos waste site being conducted by EPA's Region V Office, it is necessary to estimate airborne concentration levels of asbestos emanating from the site.

Two previous air monitoring programs have been conducted at the site, one in 1973 and the other in 1982. Although quantitative estimates of airborne asbestos concentration levels were produced, neither program was conducted in a manner that allows the data to be evaluated objectively with respect to representativeness, accuracy, and precision.

A monitoring program has been designed based on recent EPA experience and guidelines on asbestos sampling and analysis. In addition a quality assurance (QA) plan has been developed to govern the conduct of the program. The design specifies:

1. Sampling parameters
2. Analytical methods
3. QA requirements
4. Statistical summaries and data interpretations

A brief sampling test will be conducted at the outset to confirm decisions based on evaluation of historical data regarding sampling parameters such as flow rate and duration of sampling. With the exception of potential changes resulting from the test, the highlights of the recommended program are:

1. Collection of 30 samples, 25 on-site and 5 at a background site;
2. Sampling at 5 locations on-site and at 1 background site;
3. Collection of samples on 5 days at each location;
4. Collection of 12-hour samples on each day at each on-site location and 24-hour samples on each day at the background site, at the sampling rate of 15 liters per minute;
5. Analysis of the samples using Transmission Electron Microscopy (TEM);
6. Analysis of 12 QA samples (blanks, duplicates, replicates, referee) using TEM;
7. Collection of data on wind direction and speed, and compilation of other relevant ancillary information required for QA; and
8. Documentation of all QA activities.

## I. STUDY OBJECTIVES

The Johns-Manville Corporation operates an asbestos waste disposal site in Waukegan, Illinois. The EPA Region V Office is conducting an investigation of the site to assess the degree of hazard from airborne asbestos and the need for remedial action. As part of the EPA investigation, on-site measurements of airborne asbestos concentrations will be used to estimate the extent to which concentrations are elevated compared to reference levels, and the exposure potential for residents of surrounding areas.

The objectives of the study reported here are two-fold:

- To evaluate existing asbestos measurement data at the waste site for information on the "strength" of the site as a source of asbestos fibers; and
- To specify an air monitoring plan for the collection and analysis of additional data on airborne asbestos concentrations.

Two air monitoring studies have been conducted at the waste site. The results of these studies are evaluated for representativeness, accuracy, and precision. One study also provided estimates of asbestos levels in surrounding neighborhoods based on atmospheric dispersion modeling results. These estimates are evaluated for usefulness in hazard assessment.

With respect to additional air monitoring, the specified plan contains information on all aspects of measuring airborne asbestos at the Johns-Manville site. Specific components of the plan include a sampling design, sampling instrumentation and procedures, sample analysis, quality assurance, and statistical evaluation of the results.

## II. DESCRIPTION OF THE SITE

The Johns-Manville Corporation maintains an asbestos products manufacturing plant in Waukegan, Illinois, located on the Lake Michigan shoreline. A landfill for the disposal of asbestos waste material is adjacent to the manufacturing facility. The landfill occupies about  $0.48 \text{ km}^2$  (120 acres) and features (1) a pit 15 m (50 feet) deep and 46 m (150 feet) in diameter where friable waste is deposited, (2) an area of about  $0.14 \text{ km}^2$  (35 acres) where most of the dry material has been deposited, and (3) a series of lagoons delineated by berms composed of dry landfill material (see Figure 1). On-site waste consists of both friable and nonfriable materials. Although only friable materials continue to be dumped (total friable waste averages about 100 kilograms per month), previously disposed nonfriable material appears to have degraded over time. A thick coating of light-colored dust covering the entire site is evidence of material degradation. (Most of the nonfriable waste is white or light-colored.)

The waste disposal operation involves filling, transporting, and emptying an enclosed container in the bottom of the 15 m pit. The waste is then covered immediately with soil. Asbestos could become airborne at various points in this process, the most obvious being during the container dumping procedure. The application of soil cover could also suspend fibers. However, suspension of degraded materials (light-colored dust) by the wind would appear to be the major source of airborne fibers.

## III. MEASURING AIRBORNE ASBESTOS

A short discussion of methods for measuring airborne asbestos will prove useful in setting the context for both the evaluation of previous air monitoring at the Johns-Manville site, (Section IV) and recommendations for additional air

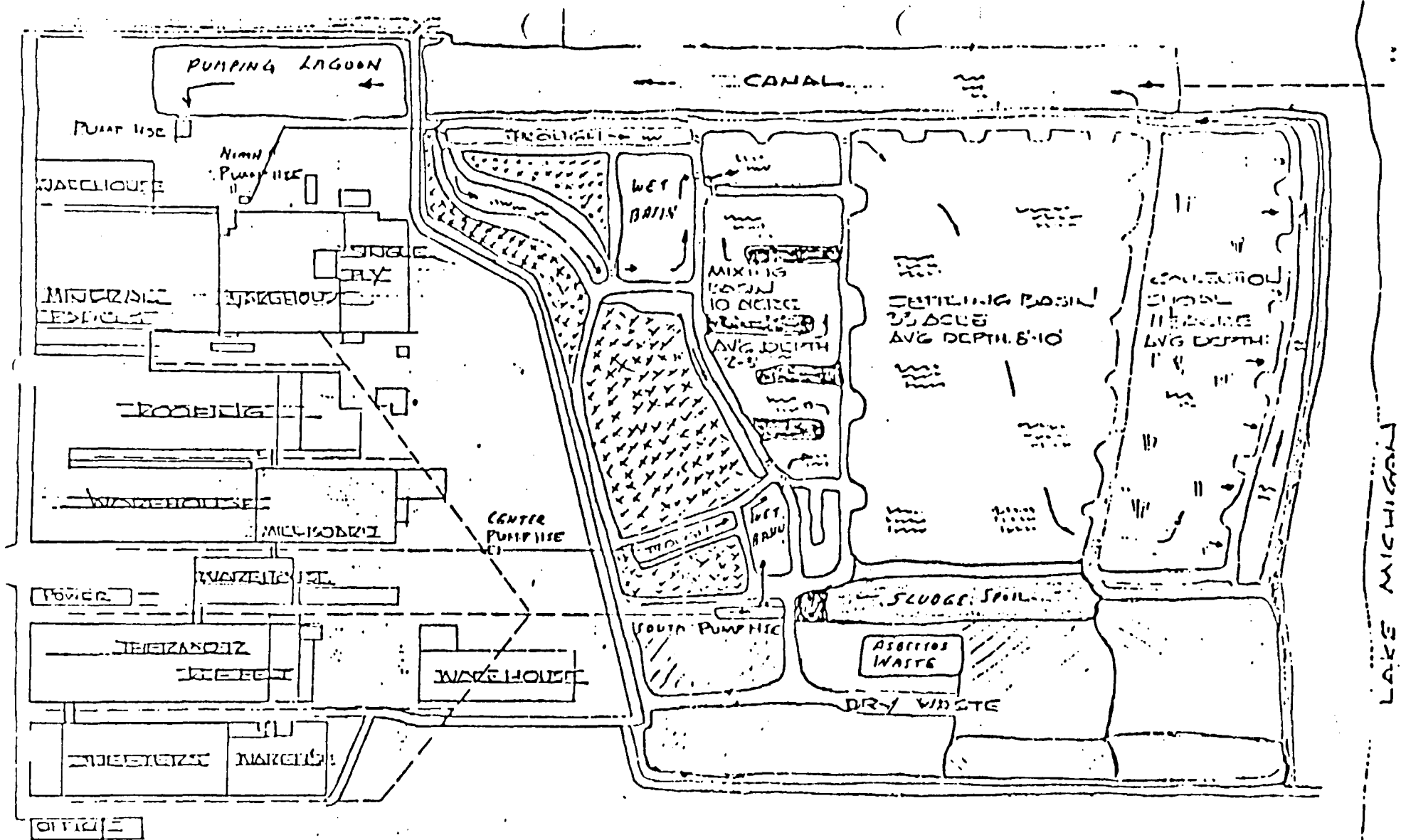


Figure 1. Johns-Manville asbestos manufacturing and waste disposal site, Waukegan, Illinois.

monitoring (Section V). Specifically, conclusions regarding the usefulness of existing air monitoring data and specifications for additional monitoring will reflect the utility and limitations of sampling and analysis procedures.

Airborne asbestos fibers are collected by drawing a measured volume of air through a filter which traps the fibers. The collected fibers are then examined microscopically and those on a small section of the filter are counted and sized. The concentration of fibers in the air from which the sample was taken is calculated based on the number and dimensions of fibers counted, the size of the filter area examined, and the total volume of air sampled. Concentrations are expressed either as fiber counts (fibers per cubic meter [ $f/m^3$ ]) or fiber mass (nanograms per cubic meter [ $ng/m^3$ ]).

Two general microscopic methods have been employed to examine asbestos fibers--one based on light microscopy and one based on electron microscopy. More specifically, the light microscopic method employs a phase contrast microscope (PCM) and the electron microscopic method typically employs a transmission electron microscope (TEM).\*

The PCM technique has been used for many years to determine compliance with the Office of Health and Safety Administration (OSHA) standard for exposure to asbestos fibers in the industrial workplace. Measurement using PCM is an inexpensive method but has several fundamental limitations. First, PCM is unable to distinguish asbestos from nonasbestos fibers; instead, all fibers are counted. Second, PCM cannot detect very thin fibers--those less than about  $0.3 \mu m$  in diameter--and the standard measurement protocol is designed to count only those fibers longer than  $5 \mu m$  in length. As a result, PCM measurements can be highly misleading where (a) many nonasbestos fibers are present, or (b) asbestos fibers have dimensions below the stated limits.

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\* Scanning electron microscopes have also been used, primarily on an experimental basis.



The alternative method based on TEM overcomes limitations associated with PCM. The higher magnification capabilities of the electron microscope allow fibers as thin as 0.01  $\mu\text{m}$  to be detected. (Counting protocols usually specify that only fibers with at least a 3:1 aspect [length-to-width] ratio should be counted.) In addition, chemical and/or crystallographic analyses are typically made on fibers observed by TEM to establish whether or not they are asbestos. The major disadvantage of using TEM is its high cost and the fact that few laboratories are qualified to perform TEM asbestos analysis.

A less significant limitation of TEM involves sample preparation procedures. If a filter is contaminated with organic materials to the extent that these materials would interfere with asbestos fiber examination, then the filter should be combusted (ashed) and the remaining material refiltered before microscopic examination (USEPA 1978). Ashing and refiltering will break fibers and, as a consequence, distort the meaning of fiber counts. (For this reason, fiber concentration measurements are typically expressed in mass units [ $\text{ng}/\text{m}^3$ ]) when these sample preparation procedures are employed.) Ashing and refiltering also runs the risk of destroying or losing some fibers.

Another problem which applies to both PCM and TEM methods involves measuring aggregations of fibers. Since counting and sizing individual fibers which comprise bundles or clumps is not possible, the true concentration of asbestos in mass units will be underestimated to the extent that airborne fibers are present as aggregates.

Comparing the limitations of both PCM and TEM, EPA now recommends that TEM be used to measure asbestos fibers collected from the ambient air and in all nonindustrial indoor settings. However, PCM is still used by many to measure asbestos levels in nonindustrial as well as industrial settings. The attraction of PCM appears to be the perceived ability to compare PCM results with the OSHA exposure standard. However, the OSHA standard was

designed for application in the asbestos industry where many if not all fibers measured by PCM are asbestos. As noted above, all fibers measured are not necessarily asbestos in other settings. In addition, the current OSHA standard was set to protect against asbestosis only. It does not appear to be stringent enough to protect against asbestos-induced cancer.\* Thus, the comparison of total fiber concentrations as measured by PCM in nonindustrial settings with the OSHA standard is not likely to be meaningful.

#### IV. EVALUATION OF AVAILABLE DATA ON LEVELS OF AIRBORNE ASBESTOS AT THE SITE

Levels of airborne asbestos fibers have been monitored twice at the Johns-Manville asbestos waste site, once in 1973 and once in 1982. In addition, levels in a broad area surrounding the site were estimated with an air quality model based on the 1973 monitoring results. The evaluation of these studies that follows consists of a characterization of the methods, and an assessment of the results for representativeness accuracy, and precision.

##### A. The Illinois Institute of Technology Research Institute Study

The Illinois Institute of Technology Research Institute (IITRI) conducted a brief monitoring and modeling study of the Johns-Manville site in December 1973 (USEPA 1974). This was part of a larger EPA-sponsored study of asbestos emissions from open sources.

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\* As of July 1, 1976, the OSHA standards were set at 2 f/cm<sup>3</sup> (2 million f/m<sup>3</sup>) averaged over 8 hours and a ceiling level not to exceed 10 f/cm<sup>3</sup> "at any time". OSHA is now evaluating the effect of lowering the 8-hour standard to either 0.5 or 0.1 f/cm<sup>3</sup> in order to protect workers against cancer, as published in the FEDERAL REGISTER (47 FR 1807).

## 1. Description and Results of Air Monitoring

Monitoring was conducted on December 8, 1973, at three on-site locations (see Figure 2). Specifications of the sampling and analysis procedures are shown in Table 1. Samples were analyzed by both phase contrast microscopy (PCM) and transmission electron microscopy (TEM). Environmental conditions during the sampling period were as follows: air temperature--approximately 0°C (32°F), wind direction--south to southwest, and wind speed--4.5 to 6.7 m/sec (10-15 mph). Operations at the site were continuous, with waste dumped from open trucks down the slope of a 9 m (30 ft) high pile. Waste consisted of broken asbestos-cement pipe, floor tile, asbestos paper and building board, and waste from the settling ponds and baghouses. The top surface of the pile was covered with soil, presumably on a daily basis.

The results of the air monitoring study are summarized in Table 2.

## 2. Description and Results of Air Quality Modeling

An atmospheric dispersion model was employed to estimate the impact of fiber release at the Johns-Manville waste site on air levels in surrounding areas. The air monitoring results at the site were used to estimate asbestos emission rates and local meteorological conditions were used to estimate transport of fibers from the site. The highest levels estimated were for the nearest residential neighborhoods--8 fibers/m<sup>3</sup> based on PCM air monitoring results and  $2 \times 10^6$  fibers/m<sup>3</sup> based on TEM monitoring results.

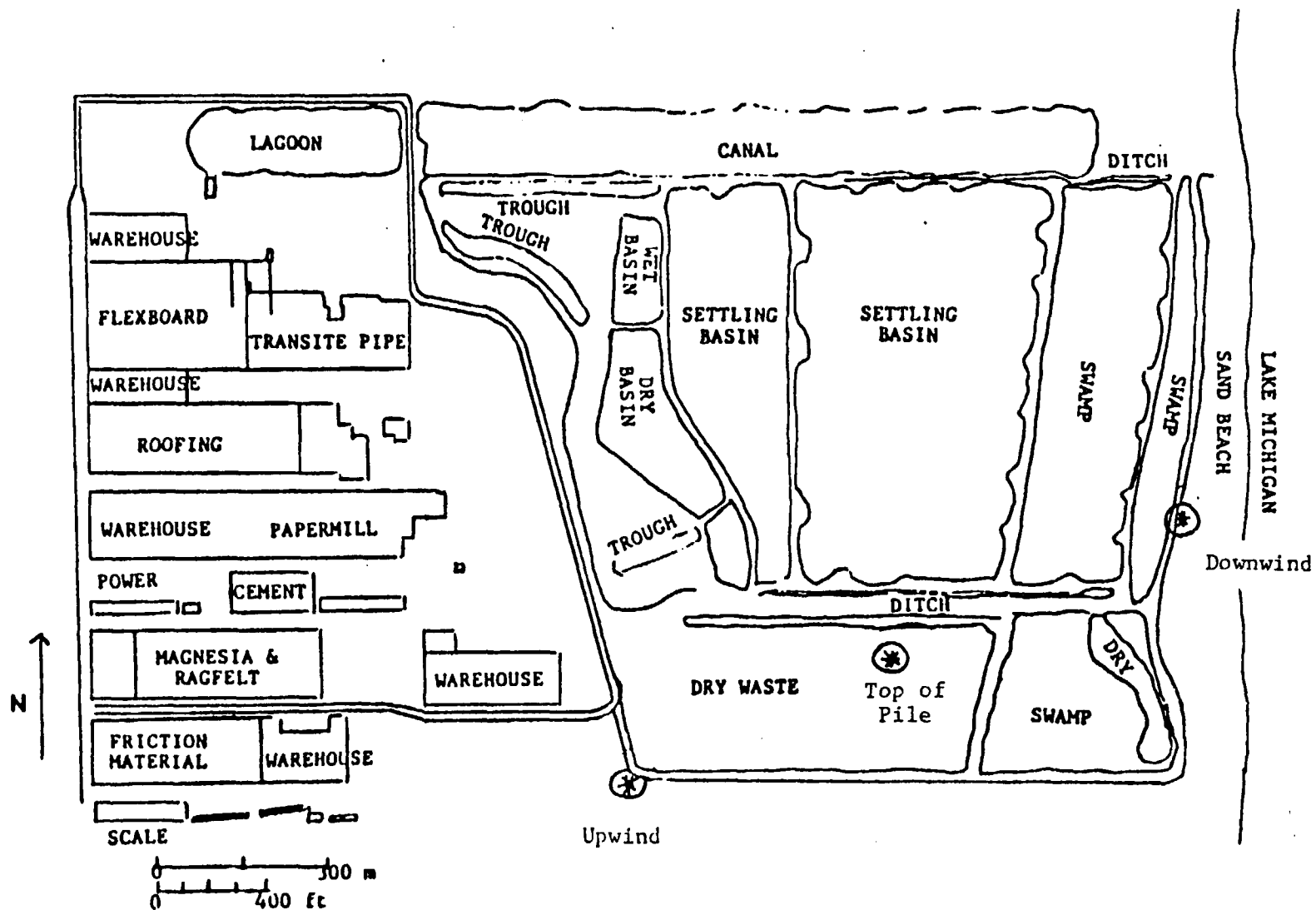


Figure 2. Monitor locations in the IITRI study.

Table 1. Specifications of IITRT Air Monitoring Study

Sampling instrument	Type of filter	Sampling time	Sample volume	Sample preparation	Analytical instrument	EM magnification
Not noted	Millipore with 0.8 $\mu\text{m}$ pore size (filter material not noted)	At least 3 hours	97 - 156 $\text{m}^3$	Filter dissolved in acetone, fibers deposited on carbon substrate of EM grid	PCM and TEM, no crystalline or chemical analysis of fibers	TEM:16,000x; fiber resolution = 0.020 $\mu\text{m}$ diameter  PCM:500x

Table 2. Results of the IITRI Asbestos Monitoring Study  
of the Johns-Manville Asbestos Waste Site, 1973

<u>Monitor Location</u>	<u>Fiber Concentration (f/m<sup>3</sup>)<sup>a</sup></u>	
	<u>PCM<sup>b</sup></u>	<u>TEM<sup>c</sup></u>
Upwind	$1.7 \times 10^2$	$5 \times 10^7$
Top of Pile	$1.6 \times 10^2$	$7.5 \times 10^7$
Downwind	$2.3 \times 10^3$	$4.3 \times 10^7$

<sup>a</sup> These are the results of measuring the fibers on a single filter at each location. Sampling and analysis specifications are shown in Table 1. Monitor locations are shown in Figure 2.

<sup>b</sup> Measurements made with a phase contrast microscope.

<sup>c</sup> Measurements made with a transmission electron microscope.

### 3. Evaluation of Monitoring and Modeling Results

The IITRI monitoring results are problematic for the following reasons:

- The results may reflect significant bias since the ground was reported to be frozen at the time of sampling, thus greatly reducing the tendency for fibers to become airborne;
- Rain during the day before sampling commenced may have cleansed the air of many fibers;
- Too few samples were collected (only three locations and one time period) to capture the expected spatial and temporal variation in levels of airborne asbestos;
- No quality assurance measures were employed (at least none are reported). Thus no judgement can be made regarding the accuracy of the results.
- Descriptions of sampling instruments and procedures are too sketchy to judge the adequacy of sampler flow rate and filter type; and
- No attempt was made to identify the mineral content of the fibers. Thus, no estimate of asbestos concentrations can be made.

The air quality modeling results can be questioned on the same grounds, since the estimated asbestos emission rate for the waste site was derived from the monitoring results. Also, additional uncertainty is introduced by the use of meteorological data from the local airport since on-site data were not available.

### 4. Conclusions

The IITRI study provides very little useful information on levels of airborne asbestos at the Johns-Manville site. Apart from the fact that the study is 10 years old and site conditions have changed appreciably, fundamental problems with

sample collection and analysis render the results of little value even as reference points. As extensions of the monitoring results, the air quality modeling estimates are similarly flawed.

B. The <sup>Ecology</sup>~~Energy~~ and <sup>Environment</sup>~~Ecology~~, Inc. Study

Air monitoring of the Johns-Manville's site was conducted on April 28, 1982, by <sup>Ecology</sup>~~Energy~~ and <sup>Environment</sup>~~Ecology~~, Inc. (EEI), under contract to the USEPA. The samples collected were analyzed using TEM both by an independent laboratory (EMS Labs, Inc.) and by the Johns-Manville Corporation. The EEI/EMS results were also reviewed by scientists retained by the Centers for Disease Control (U.S. Department of Health and Human Services).

1. Description and Results of Air Monitoring

Sampling was conducted during a single day at the three locations shown in Figure 3. The temperature was 4.5°C (40°F), winds were from the northwest at 4.5-6.7 m/sec (10-15 mph), and no rain fell during or 24 hours before sampling. Sampling and analysis specifications are shown in Table 3.

The results of the EEI study are shown in Table 4. Analytical measurements made by both EMS and Johns-Manville laboratories are included. The concentration values shown in Table 4 are weighted averages of two samples collected at each monitor. (The dichotomous sampler employed by EEI separates the particles collected into two size fractions by aerodynamic diameter [less than 2.5 µm and 2.5 - 15 µm]). As noted in a June 30, 1983, memo from Johns-Manville to the EPA Region V





Table 3. Sampling and Analysis Specifications of EEI Air Monitoring Study

Sampling instrument	Flow rate	Sampling time	Type of filter	TEM analysis--sample preparation and magnification		Asbestos identification	
				EMS <sup>b</sup>	J - M <sup>c</sup>	EMS <sup>b</sup>	J - M <sup>c</sup>
Dichotomous sample	1.67 and 15.03 lpm <sup>a</sup>	7.5 Hours	Cellulose ester membrane, 0.8 $\mu$ m pore size	Ashing and re-filtering on 0.1 $\mu$ m nucleofilters deposition on EM grids using modified Jaffe Wick method; 20,000x magnification; 2 - 20 fields examined	"USEPA recommended methodology"; 20,000x magnification; at least 10 fields examined	Electron diffraction to identify asbestos	Electron diffraction and energy dispersive x-ray analysis to identify asbestos

<sup>a</sup> These are the flow rates for the coarse and fine mode collection chambers, respectively.

<sup>b</sup> EMS - EMS Laboratory

<sup>c</sup> J-M - Johns-Manville Corp.

Table 4. Results of the EEI Asbestos Monitoring Study at the Johns-Manville Asbestos Waste Site, 1982

<u>Monitor location</u>	<u>Asbestos concentration (ng/m<sup>3</sup>)<sup>a</sup></u>	
	<u>EMS<sup>b</sup></u>	<u>J - M<sup>c</sup></u>
Upwind	5	-- <sup>d</sup>
Midsite	288	167
Downwind	189	24

<sup>a</sup> These are the results of measuring asbestos fibers on a single filter at each location. Transmission electron microscopy was the measurement method. Sampling and analysis specifications are shown in Table 3. Monitor locations are shown in Figure 3.

<sup>b</sup> EMS Laboratory results.

<sup>c</sup> Johns-Manville results.

<sup>d</sup> J-M believes the filter was damaged.

Waste Management Division, the correct way to calculate total sample concentrations is to weight the results for the "fine" and "coarse" fractions by the amount of air sampled.\*.

## 2. Evaluation of Results

The EEI air monitoring study is an improvement over the IITRI study in a number of areas. First, the meteorological conditions were more conducive to observing wind-generated airborne asbestos. Conditions were dry with temperatures above freezing and wind velocities were substantial. Second, the samples were analyzed by TEM and the fibers were identified as asbestos. Moreover, the analysis of the same filters by two independent laboratories (EMS and Johns-Manville) provides some information on the reliability of the measurements.

One important deficiency of the EEI study involves the number of samples drawn and the location of the monitors. Since the goal of the monitoring program is to gauge the quantity of asbestos fibers released over the entire site by measuring the on-site levels of airborne asbestos, air samples should be taken at various points within the site and at various times over an extended sampling period to capture the spatial and temporal variation in asbestos concentrations. Three sampling locations and one 7.5-hour sampling time are not sufficient to produce acceptably accurate and precise estimates of air concentrations.

A second deficiency is the lack of sampling in a "background" location. The significance of the asbestos concentrations on-site should be judged, in part, by comparison to measured air levels at a site unaffected by the Johns-Manville waste site or any other source of asbestos

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\* Due to the design of the dichotomous sampler, the flow rate through the chamber which entrains the larger particles is about 10% of the flow associated with the smaller particles.

fibers. The selection of a "background" site, and the sampling design (duration and frequency of sampling) implemented there are critical considerations. EEI apparently selected the upwind monitor located slightly off-site to represent background concentrations. However, this location is much too close to the waste site to satisfy the criteria for a background site.

### 3. Conclusions

The results of the EEI study suggest that levels of airborne asbestos may be elevated at the Johns-Manville waste site. This conclusion assumes that asbestos concentrations at background sites in Waukegan approximate typical levels observed at urban background sites elsewhere (1-10 ng/m<sup>3</sup>) (Nicholson 1971). Additional air monitoring both on- and off-site will be necessary to confirm this preliminary conclusion.

## V. PLAN FOR ADDITIONAL MONITORING

Specifications for a new air monitoring study are presented in this section. Included are discussions of air sampling, sample analysis, quality assurance procedures, and data interpretation.

### A. Sampling Plan

As discussed in Section I, the purpose of air monitoring is to estimate levels of airborne asbestos at the Johns-Manville site and to compare them with levels at sites which are not influenced by disposal site activities or other sources of asbestos. This requires estimation of both average concentrations and the variability of measured levels at each site. The sections which follow describe considerations for selecting (1) the background site, (2) the number of samples required for various levels of precision in the measurements,

(3) the location of monitors at each site, and (4) the sampling times and volumes. The final section describes sampling instrumentation and procedures.

#### 1. Background Site Selection

A desirable location for a background site is one far upwind from the waste disposal site. Given the expected predominance of winds from the east, west, northeast, and southwest (and thus the low probability of northerly winds) due to lake/land effects at the Johns-Manville site,\* a location to the south of the plant should be sought for a background site. To assure minimal influence from the waste site, a distance of at least 5 km is recommended. The site itself should be a relatively homogeneous area in terms of land use, and should not be influenced by any other source of asbestos.

Of particular importance is the location of tire stores or automobile repair shops where brakes are repaired. Since asbestos is frequently used in brake materials, brake repair operations may be a significant source of airborne asbestos.

Sites near gravel or dirt roads should also be avoided for two reasons. First, these sites may be very dusty and, thus, overloading of collection filters may become a problem. Second, some communities have used asbestos-containing crushed stone for road paving. Traffic on these roads may suspend asbestos fibers.

Any data on airborne asbestos from previous air monitoring studies in the Waukegan area should be used in selecting a background site. Low measurements near candidate sites would confirm their suitability.

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\* Prevailing annual wind patterns at a local airport are NE-SW. A lake-side location should accentuate this pattern and further minimize northerly winds.

## 2. Number of Samples

The number of samples needed for a desired level of precision in the results depends on the magnitude of the variability associated with all phases of the sampling and analysis process. If several air samples are taken in the same general area but at slightly different locations (e.g., at different points within the waste disposal site) or at different times at the same location, the measurements of sampled material will differ from one another. These differences constitute the sampling component of variability. Sampling variability is due to random fluctuations in the population being sampled, and to factors such as wind speed and direction, atmospheric stability conditions, and the distance from emission sources such as dumping activities or roadways. These latter factors may be viewed as systematic influences on sampling variability, and potentially can be controlled through sample design.

A second type of variability is that associated with the air sampling instrumentation and chemical analysis procedures. This is called analytic variability and is especially important for asbestos since asbestos fibers are difficult to detect and characterize. This variability can be further subdivided into variability between laboratories and variability within laboratories. Variability between laboratories is due to differences in types of equipment, interpretation of procedures, and analytical practices; variability within laboratories is due to differences between individual analysts (based on differences in experience and training) and differences between repeated readings obtained from the same sample by a single analyst as a result of variability in preparing a sample and in counting fibers.

Due to the sources of variability enumerated above, the measured concentration of asbestos in a single air sample collected at one location for a short period of time is unlikely to be equal of the "true" concentration averaged over the entire

site and for a longer time. The degree to which a single estimate departs from the "true" value is called the estimation error. This error can be reduced by forming an average of samples taken at more locations, at more times, and by repeated measurement in the laboratory. The magnitude of error will depend both on the number of samples and the total sampling and analytic variability of the measurements.

In order to calculate the number of samples required to achieve a desired estimation error, the amount of expected variability in the measurements must be approximated or assumed. Some data are available from which estimates can be made of variability associated with the analytical method (between and within laboratories), but the spatial and temporal variability of airborne asbestos at the Johns-Manville site is unknown. Therefore, required sample sizes have been calculated assuming a range of possible variabilities, where variability is measured relative to the expected concentration using a term called the coefficient of variation (standard deviation divided by the mean). A large coefficient of variation (e.g., greater than 100%) reflects a high level of variability.

Table 5 shows the relationship between the coefficient of variation, estimation error, and the number of required samples.\* For example, if the coefficient of variation for the measurements is 100%, then taking 19 samples will "assure" that the estimation error is  $\pm 60\%$  of the true mean.† In other words, the average concentration for 19 samples should fall somewhere between 60% less than and 60% greater than the true

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\* These calculations are based on several assumptions which may hold only approximately in practice. Therefore the sample sizes should be used only as a guide. See Appendix A for a discussion of the assumptions underlying the calculations.

† Although it is not possible to be absolutely sure that the true mean will fall within this interval, the probability is high. See Appendix A and footnotes to Table 5.



Table 5. The Relationship Between Sample Size, Coefficient of Total Variation, and Estimation Error

Coefficient of total variation <sup>a</sup>	Maximum acceptable estimation error as a percentage of the true mean <sup>b</sup>	Required sample size <sup>c</sup>
100%	25%	78
	50%	25
	60%	19
	75%	14
	80%	13
	100%	10
150%	25%	160
	50%	48
	60%	35
	75%	25
	80%	22
	100%	16

<sup>a</sup> Standard deviation divided by the mean and expressed as a percentage.

<sup>b</sup> Based on the 95% confidence interval for the true mean calculated from the observed data.

<sup>c</sup> The number of samples required to ensure that the estimation error is less than the specified amount in the second column, with a probability of 90%.

mean. Increasing the sample size to 25 reduces the estimation error to  $\pm 50\%$  of the true mean. Once the samples have been collected and a sample average calculated, this average becomes the best estimate of the true mean and an actual estimation error is calculated from the sample variance. (This procedure is discussed in Appendix A.)

The two coefficients of variation in Table 5 (100% and 150%) have been selected based on limited data on (1) laboratory variability in measuring asbestos, and (2) temporal variability in particulate matter concentrations at a few sites.\* Extrapolating from these data, the coefficient of total variability for airborne asbestos will likely be at least 100% and may be higher than 150%.

A minimum of 25 samples is recommended for the Johns-Manville site. This sample size would provide an estimation error of  $\pm 50\%$  of the true mean if the coefficient of variation is 100%, or  $\pm 75\%$  if the coefficient of variation is 150%.

For measurements of asbestos levels at background sites, a larger estimation error might be tolerable. For example, it may be sufficient to know only that the background concentration is less than some relatively low level, perhaps  $30 \text{ ng/m}^3$ . If the actual mean is  $10 \text{ ng/m}^3$ , then the maximum tolerable estimation error is  $\begin{matrix} -100\% \\ +200\% \end{matrix}$  (or a one-side error of +200%). A sample size of 5 would be sufficient to "assure" that the estimation error was no larger than this limit. Five samples are thus recommended for the background site.

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\* Very limited evidence suggests that the coefficient of variation in asbestos measurements due to variability between laboratories may be 50-90% (Steel, et al. 1982) and within laboratories, 30-40% (USEPA 1983). Temporal variability in 24-hour measurements of particulate matter at a sample of sites in Illinois (1980 data) produced a coefficient of variation which averaged about 45% (data from USEPA 1981).

To illustrate how the size of the estimation error influences interpretation of the monitoring results, suppose the measured mean concentration at the waste site were  $200 \text{ ng/m}^3$  with an estimation error of  $\pm 75\%$ , and the mean at this background site were  $10 \text{ ng/m}^3$  with an error of  $\pm 200\%$ . Thus, we could say (with 95% confidence) that the waste site concentration is between 50 and  $350 \text{ ng/m}^3$  and the background concentration is between 0 and  $30 \text{ ng/m}^3$ . In this example, we can be confident that the two concentrations are clearly different. The smaller the estimation errors, the easier it is to distinguish measured concentrations at the two sites.

### 3. Monitor Location

Since the air samples collected should be representative of typical concentrations at each site, they must capture both spatial and temporal variations in air levels. For the waste disposal site, five sampling locations and five sampling times are recommended, thus making a total of 25 separate samples. The sampling locations should be randomly selected within the following constraints: all locations should be at least 30-m from the boundaries of the site (to assure that measurements reflect on-site emissions), and the set of five locations should be approximately symmetrical so as to capture high concentration irrespective of wind direction or distance from on-site "sources" (e.g., the disposal pit, roadways, the main landfill). One way to select the sampling locations is to construct a transparent template with a grid superimposed on a circle with five radial sectors (i.e., each sector subscribes  $72^\circ$ ). The template is made about as large as a scale map of the waste site and placed on top of the map. The grid points on the template are numbered and a random number table used to select one location within each sector. Of course, if a selected location falls on water or another physically unsuitable spot, a

substitute must be chosen within that sector. This design is intended to make the spatial variability in asbestos concentration random.

For the background site, a single monitor operated for the same five time periods is desirable. A single monitor will suffice since temporal variability is likely to be greater than spatial variability there. The specific location of the monitor will be governed by the usual considerations of security, access, and power availability. Locations near sources of dust should be avoided to prevent overloading of filters with particulate matter.

#### 4. Sampling Times and Volumes

Based on the likelihood of day-to-day variability in on-site activity and meteorological conditions, sampling should be conducted on five separate days. Sampling periods of 12 hours for waste site monitors and 24 hours for background monitors are suggested. The start and end hours for the 12-hour sampling period should be timed to coincide with the start and end hours of the day work shift at the Johns-Manville plant. These sampling periods should smooth out hourly variability in asbestos levels. Where possible, days with different wind speed and direction should be chosen. In all cases, days with rain or days following precipitation by less than 24 hours should be avoided.

The total volume of air to be sampled is dictated by (1) the lower detection limit of the analytical methodology,\* (2) total concentrations of particulate matter at the sites (and, thus, the potential for overloading filters), and (3) accepted operating practices for sampler flow rates and filter

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\* At least 10 asbestos fibers should be counted during EM examination (USEPA 1978).

face velocities for airborne asbestos monitoring (Yamate 1982). Based on the findings of the EEI study and on other airborne asbestos monitoring studies (USEPA 1983), a total sample volume of 6,000-11,000 liters is recommended. A volume of 10,800 liters would be collected if the waste site samples were operated at a flow rate of 15  $\ell$ pm (12 hrs. @ 15  $\ell$ pm Total volume at the background site would be twice as large (21,600 liters) thus improving the likelihood of collecting a detectable quantity of asbestos.

Filter "overloading" usually refers to gross clogging of the filter media. In the context of monitoring airborne asbestos, however, it may refer to contamination of the filter with organic substances requiring that the filtered material be ashed and refiltered prior to examination by EM. Since ashing and refiltering is not the preferred treatment, a pretest of the sampling plan is recommended to test for contamination by organic material.

Ashing and refiltering is also necessary if millipore rather than nucleopore filters are used. Millipore filters are sometimes used because they retain fibers better during filter handling and transport. The pretest is designed to evaluate this suitability of nucleopore filters as well as to test for contamination by organic materials.

The pretest should consist of three monitors at a single waste site location. (The location should be one likely to produce high asbestos concentrations). The three monitors should be operated with three different flow rates: 5, 10, 15  $\ell$ pm, and the sampling time should be 12 hours. These combinations of flow rates and sampling times will produce high enough sample volumes to assure sufficient quantities of fibers for precise estimates at the highest rate (15  $\ell$ pm) and low enough filter loadings to reduce organic contamination at the lowest (5  $\ell$ pm).

After collection, the three pretest samples should be examined by the EM laboratory. Sample preparation should not include ashing and refiltering. If contamination by organic

materials is still substantial at the lowest flow rate in the opinion of the electron microscopists, or loss of collected fibers from the nucleopore filters after sampling appears to have occurred, then the use of millipore filters and ashing/refiltering procedures will be necessary. Otherwise, the highest of the flow rates which still produces tolerable contamination should be selected for the monitoring study.

## 5. Instrumentation and Sampling Specifications

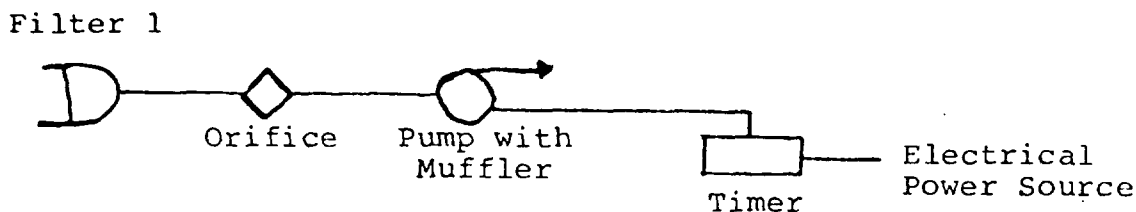
The following sampling procedures are within the class of procedures tested and recommended by EPA (USEPA 1978 and Yamate 1981). More specific information on selected procedures can be found in Appendix B.

### a. Sample Setup

The sampling system should consist of:

- A Gleman magnetic-type open-face filter;
- A critical flow orifice;
- A diaphragm pump with muffler;
- Associated plumbing and stand; and
- Timer (if desired).

The sampler setup is schematically represented as follows.



b. Specifications

- Flow rate: 5, 10, and 15 lpm for the pretest; one of the three will be selected for the study;
- Filter type: For the pretest and if organic contamination or fiber loss from the filter is not a problem: 47 mm polycarbonate nucleopore with a 0.2  $\mu$ m pore size.  
If organic contamination or fiber loss is a problem: 47 mm cellulose acetate (millipore type HA) with 0.45  $\mu$ m pore size.
- Filter height: 1.5 m

c. Sampling Protocol

1. Clean and dry filter holder.
2. Place filter in holder, assuring proper position, see filter handling section below.
3. Mount filter holder such that filter is in a vertical position (perpendicular to ground).
4. Check plumbing for any leaks.
5. Check flow with flowmeter using manual control of pump.
6. Set automatic timer to desired on-off time settings (if timer is to be used).
7. Make appropriate logbook entries.
8. Conduct sampling.
9. After sampling period, check flow.
10. Rotate filter to a horizontal position and remove. Place nucleopore or millipore filter in a petri dish for proper handling and transport.

#### d. Filter Handling

During loading and unloading of the filter holder, the filters should be handled by forceps (not with fingers). When a filter is removed after exposure, it should be placed in the petri holder exposed side up and maintained in that position during the handling and transport of samples back to the laboratory. The samples should be hand-carried to the selected TEM laboratory in a container that will keep the petri dish in a horizontal (flat) position at all times (handling, transport, and storage).

The chain-of-custody system should be followed at all times (see Appendix B). A chain-of-custody record, therefore, will be kept on each filter.

Field blanks should be randomly selected at each site and for each sampling time (see Section V. C. below). Any dropping or mishandling of a filter after collection must be recorded. Each filter holder should be labeled according to a coding system. Laboratory blanks should be selected prior to field sampling (see Section V. C. ). If possible, all filters at the same site should be from the same production lot.

#### e. Meteorological Observations

A wind vane and anemometer should be used to record wind direction and speed at the waste site. Recorded data should then be used to draw a wind rose for each day of sampling.



#### f. Logbook

An important part of any successful field program is the accurate observations and recordkeeping of the field team. At a minimum, logbook entries should include:

1. Name of field operator;
2. Date of record;
3. Number and location of site;
4. Position of sampler within site;
5. Brief description of site;
6. Corresponding filter number;
7. Sample flow rate at start of sampling period;
8. Start time;
9. Stop time;
10. Sample flow rate at end of sampling period;
11. Wind rose for the sampling period;
12. Description of meteorological conditions; and
13. Comments.

#### B. Sample Analysis

Air samples should be analyzed by transmission electron microscopy according to the methodology recommended by EPA (USEPA 1978 and Yamate 1981). Two alternative sample preparation protocols are employed. The first is utilized when the sample is collected on polycarbonate nucleopore filters and, thus, when contamination by organic materials is not a problem. The second protocol is employed when the sample is collected on millipore filters (typically cellulose ester or acetate). Which protocol is employed will be determined by the outcome of the pretest, as discussed previously. Brief descriptions of the two protocols are provided below; detailed sample analysis instructions appear in Appendix C.

##### 1. Sample Preparation

###### a. Samples on Nucleopore Filter

When nucleopore filters are used, the filter is first coated with a carbon film using a vacuum process. The

coated sample is then transferred to an EM grid using a modified Jaffe washer technique. In essence, the nucleopore filter is placed on top of a carbon-coated EM grid and the filter is dissolved with chloroform. This deposits the carbon-coated sample directly on the grid.

#### b. Samples on Millipore Filters

Samples on millipore filters must be ashed and then refiltered on a nucleopore filter. The filters are first ashed at low temperatures to destroy the filter medium and any organic contaminants. The ashed residue is then redispersed by ultra-sonification and filtered with a nucleopore filter.

### 2. EM Examination

Fibers are scanned, counted, and sized using an electron microscope at 20,000X magnification. Asbestos fibers are identified using selective area electron diffraction (SAED) analysis.

#### C. Quality Assurance

To ensure that the information obtained from the air monitoring study is reliable, a quality assurance (QA) program is needed. A formal QA plan is developed to establish organizational responsibilities and to specify procedures for implementing the plan. A complete QA plan is described in Appendix D; only the names of the team members need to be added. The key objectives and elements of the plan are briefly described below.

## 1. Documentation

Once completed, the QA program provides documentation of all procedures and activities. Such documentation raises the confidence of everyone associated with the study, especially potential users of the study results. Documentation also allows the handling and treatment of individual samples to be traced, if this is needed.

## 2. Corrective Action

A QA program will provide a mechanism for taking corrective action in response to the identification of data problems. Ideally, corrective action will be taken quickly enough to hold the loss of data to a small fraction of the entire data set.

## 3. QA Checks

A QA program establishes a series of checks to detect gross problems with data collection, handling, and analysis procedures. These include the analysis of blank samples, multiple analyses of single samples within a laboratory, and multiple analyses by more than one laboratory.

### a. Field and Laboratory Blanks

During each sampling period and at each sampling site (i.e., waste disposal and background sites), at least one filter should be randomly selected as a field blank from the filter supply. Thus, a total of 10 field blanks is needed for this study. The blank filter is labelled and handled as any other filter but is not actually used for air sampling. A proportion of the field blanks (at least three) are submitted for analysis along with the test filters. The field blank

provides a check for possible filter contamination. If contamination appears to be a possibility, additional field blanks can be analyzed to help determine the extent of the problem.

In a similar manner, at least three blank filters should be exposed on a laboratory bench during preparation and analysis of the samples. At least one of these is then analyzed to check for contamination in the laboratory.

#### b. Replicate and Duplicate Filter Analysis

As a means of quantifying analytical variability due to preparation and counting procedures, some filters should be selected at random for replicate analysis and some for duplicate analysis. Replicate analyses are done using two independent preparations from the same filter. Duplicate analyses are done by two different analysts using the same TEM grid preparation. It is recommended that a minimum of three filters be selected for each type of analysis and that further analyses be conducted if serious discrepancies appear. For this reason, it is important that all filters and sample preparations are carefully stored.

#### c. Interlaboratory Quality Assurance

A proportion of the filters (usually about 10% or three for this study) should be analyzed by a second laboratory. These filters are selected at random from the test filters and each is divided in half. One half is analyzed by the main laboratory and the other half by the second laboratory. If serious discrepancies appear, additional filters should be analyzed.

#### D. Statistical Evaluation

The data will be used to estimate a mean airborne asbestos concentration for the Johns-Manville waste disposal site and for the background site.\* For each mean, a 95% confidence interval will be obtained to provide a measure of the estimation error. Comparisons between disposal site and background air levels can be made using standard statistical methods.

After the data have been collected and an estimate of variance is available, it is possible to evaluate the power of the statistical tests. In the case in which no statistically significant difference is found between two estimated means, the power calculation will provide a measure of how much confidence one can have in that conclusion.

The results from the various QA samples (field blanks, external laboratory, replicate, and duplicate samples) will be compared with the appropriate original analyses. The small number of QA samples precludes formal statistical analysis. However, if inconsistencies or large discrepancies are observed, further QA samples can be analyzed since only a portion of each filter is needed for each analysis.

#### E. Summary of Sampling and Analysis Design

Table 6 summarizes the key elements of the recommended air monitoring program.

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\* Averages could also be estimated for subareas within the waste site, but the confidence intervals for these estimates would be very large due to the small number of samples. Data on wind direction and speed will be used to judge the representativeness of the asbestos measurements for each site.)

Table 6. Summary of Key Elements of New Air Monitoring Study

Site	Number of monitors	Sampling time	Flow Rates		Type of Filter		EM Sample Preparation	
			Pre-test	Study	Pre-test	Study	Pre-Test	Study
Waste	5	5 days @ 12 hrs/day	5, 10, & 15 lpm	5, 10, or 15 lpm <sup>a</sup>	Nucleopore	Nucleopore or Millipore <sup>b</sup>	Carbon coating only	Carbon coating only or preceded by ashing & re-filtering <sup>c</sup>
Background	1	5 days @ 24 hrs/day	--	5, 10, or 15 lpm <sup>a</sup>	--	Nucleopore or Millipore <sup>b</sup>	--	Carbon coating only or preceded by ashing & re-filtering <sup>c</sup>

- <sup>a</sup> Depends on results of the pre-test, 15 lpm recommended unless a lower rate eliminates contamination by organic materials.
- <sup>b</sup> Use nucleopore filters if organic contamination is not a problem (based on results of pre-test); otherwise, use millipore filters.
- <sup>c</sup> Use ashing and refiltering procedures if millipore filters are used.

F. Cost and Time Estimate

The air monitoring study should cost between \$55,000 and \$65,000. The estimated time to complete the study is 3-1/2 months. Cost components are shown below.

- Sample collection

2 staff x 12 hrs/day x 10 days @ \$65-70/hr = \$16,000 - 17,000

- Sample analysis

45 samples @ \$600-700 = 27,000 - 32,000

- Quality Assurance and Reporting

12,000 - 16,000

Total \$55,000 - 65,000

3  
"

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## Appendix A. Calculating Sample Sizes

The term "estimation error", as used in Section V. A.2, refers to half of the length of the 95% confidence interval for the true mean. This confidence interval will be calculated from the data after they have been collected and will indicate the magnitude of the error associated with the estimation of the true mean. If the coefficient of total variation is small and/or the sample size is large, then the confidence interval will be short and one will be confident that the true mean is not very different from the value estimated from the data. By "confident" it is meant that 95% of the time the procedure for calculating a 95% confidence interval results in an interval which actually includes the true mean.

The formula for the 95% confidence interval is:

$$\bar{x} \pm t_{(0.025, n-1)} \sqrt{s^2/n}$$

where  $\bar{x}$  and  $s^2$  are the calculated sample mean and sample variance, respectively, and  $t_{(0.025, n-1)}$  is the upper 2.5 percent point of the t distribution with  $n-1$  degrees of freedom. Note that

$t_{(0.025, n-1)} \sqrt{s^2/n}$  is the estimation error. The aim is to choose the sample size  $n$  so that  $t_{(0.025, n-1)} \sqrt{s^2/n}$

is not too large. Suppose it is decided that this quantity should be no larger than  $d\mu$  where  $\mu$  is the true mean and  $d$  is a fixed proportion. For example, if the estimation error is required to be no more than 60% of the mean, then  $d$  would be made equal to 0.6. Then  $n$  has to be chosen so that

$t_{(0.025, n-1)} \sqrt{s^2/n}$  is less than  $d\mu$ .

It is not possible to be absolutely sure that for a given sample size the resulting confidence interval is sufficiently small, but it is possible to attach a probability to the chance that it will be. For example, it is possible to find  $n$  such that the probability that the confidence interval is sufficiently small is 0.9 or 0.95, or any other desired level. If the desired level is  $1-\beta$  then it is necessary to find  $n$  such that

$$P \left( t_{(0.025, n-1)} \sqrt{s^2/n} \leq d\mu \right) = 1-\beta.$$

This is equivalent to

$$P \left( \frac{(n-1)s^2}{\sigma^2} \leq \frac{(n-1)nd^2\mu^2}{\sigma^2(t_{0.025,n-1})^2} \right) = 1-\beta$$

If it is assumed that the  $n$  samples are independent observations from a normal distribution with mean  $\mu$  and variance  $\sigma^2$  then  $(n-1)s^2/\sigma^2$  has a  $\chi^2$  distribution with  $(n-1)$  degrees of freedom. The problem is thus reduced to finding  $n$  such that

$$\frac{(n-1)nd^2\mu^2}{\sigma^2(t_{0.025,n-1})^2} = \chi_{n-1}^2,$$

where  $\chi_{n-1}^2$  is the upper  $(100\%)\beta$  percentage point of the  $\chi_{n-1}^2$  distribution. Substituting  $\sigma^2 = c^2\mu^2$  gives

$$n = \left( 1 + \sqrt{1 + 4(t_{0.025,n-1})^2 (c/d)^2 \chi_{n-1}^2} \right) / 2$$

which can be solved by trial and error.

Table A-1 shows the values of  $n$  for different values of the coefficient of variation ( $c$ ), the size of the 95% confidence interval (estimation error) and different values of the probability of obtaining an error as small or smaller. For example, if the coefficient of variation is 100% and one wants to ensure with probability 0.95 that the estimation error is no greater than  $\pm 50\%$  of the true mean, then 27 samples are required. If only 22 samples are collected then the probability is reduced to 0.8.

Table A-1. Sample Size Required to Estimate the Mean with a  
Desired Level of Precision with the  
Coefficient of Variation Set at 100% and 150%

Coefficient of variation = 100% <sup>a</sup> Maximum acceptable estimation error (%) <sup>b</sup>	Probability of achieving acceptable estimation error		
	<u>0.8</u>	<u>0.9</u>	<u>0.95</u>
25	73	78	81
50	22	25	27
60	17	19	20
75	13	14	15
80	12	13	14
100	9	10	11

Coefficient of variation = 150% <sup>a</sup> Maximum acceptable estimation error (%) <sup>b</sup>			
	<u>0.8</u>	<u>0.9</u>	<u>0.95</u>
25	154	160	176
50	44	48	50
60	32	35	38
75	22	25	27
80	21	22	24
100	15	16	17

<sup>a</sup> Standard deviation divided by the mean and expressed as percentage.

<sup>b</sup> The length of the 95% confidence interval for the true mean  
calculated from the observed data.

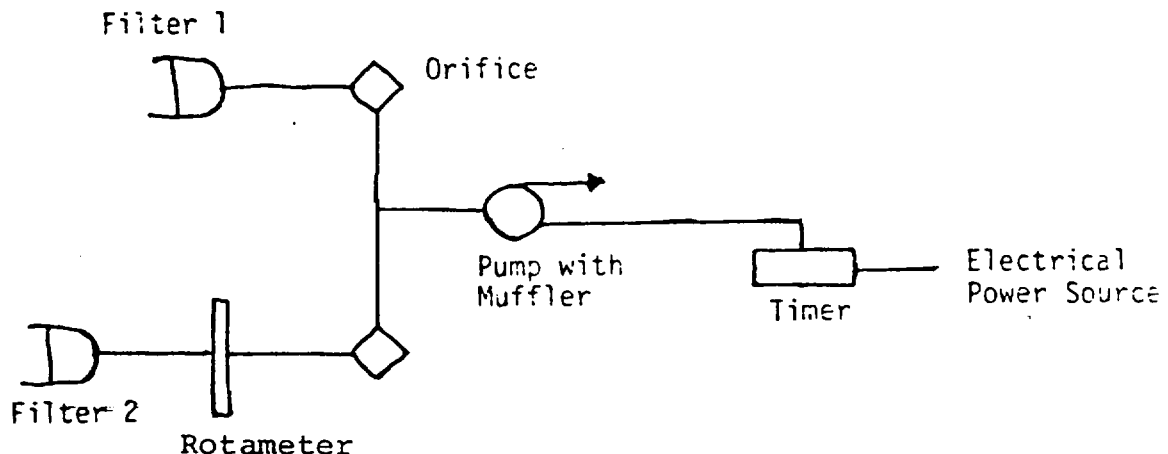
## Appendix B. Details of Selected Sampling and Analysis Procedures

### B.1 Procedure for Measuring Flow in the Field

This procedure describes the process used to determine the sample flow rates through the filters used to collect asbestos fibers in ambient air.

#### Flow Measurement

1. Set up the sampling system as shown below with the rotameter in one leg of the sampler.



2. Turn on the pump and with both filters in place, record the rotameter reading in the notebook.
3. Turn off the pump and transfer the rotameter to the other leg of the sampler.
4. With both filters in place, turn on the pump and again record the rotameter reading for the second leg.
5. Turn off the pump and remove the rotameter from the sampler.
6. Reconnect all tubing.
7. The sampler is ready to operate.
8. Procedures 1 through 5 must be repeated at the end of the sampling period.

## Calculations

1. Using the calibration curve for the rotameter, determine the flow rates for each rotameter reading and record these values on the data sheet.
2. Calculate the average flow rate for the sampling period using the following equation:

$$\text{average flow rate} = \frac{(\text{initial flow rate} + \text{final flow rate})}{2} .$$

3. Calculate the actual volume of sample collected by multiplying the average sample rate by the sampling time.

## B.2 Sample Custody

Traceability procedures described herein will be used to ensure sample integrity.

1. Each sample (filter) will be issued a unique project identification number. This number will be recorded in a logbook with the following information:
  - Name and signature of field operator;
  - Lot or assigned batch number (or any other identifiable number);
  - Filter type (e.g., millipore, nucleopore);
  - Date of record;
  - Number and location of site;
  - Position of sampler within site;
  - Use of filter, i.e., field blank, lab blank or test filter;
  - Condition of sample;
  - Sample flow rate at start of sampling period;
  - Start time;
  - Stop time;
  - Sample flow rate at end of sampling period; and
  - Any specific instructions/comments.
2. The samples will be hand-carried to the laboratory doing the chemical analysis where the package contents will be inventoried against the traceability packing slip.
3. A copy of the inventory sheets will be sent to the QA manager. The original will remain with the field sampling leader's files. Warning labels (if appropriate) will be affixed.

4. In order to maintain traceability, all transfer of samples is recorded in an appropriate notebook (where appropriate). The following information will be recorded:

- The name of the person accepting the transfer, date of transfer, location of storage site, and reason for transfer,
- The accepting party must verify transfer by initialing after his or her name; and
- The assigned sample code number remains the same regardless of the number of transfer.

After the samples are properly logged-in they will be placed in storage areas accessible only to authorized personnel. These areas will be identified as to the hazard they present. Activities will be restricted to a minimum in the storage area. Mixing, formulating, and diluting, are expressly forbidden.

## Appendix C. Analytical Protocol for Air Samples

### C.1 Sampling Handling

Select one filter from each box of 24 0.45  $\mu\text{m}$ , 47-mm Millipore HA membrane filters or 0.20  $\mu\text{m}$ , 47-mm polycarbonate Nucleopore filters to serve as a laboratory blank. Use all filters from the same production lot number, if possible. Prior to field sampling, determine if the laboratory blank filters are asbestos free by transmission electron microscope examination. Record filter box and lot number.

Upon receipt of filters from the sampling team, record in a laboratory logbook the sample numbers, date they were received, and any macroscopic identifying characteristics of particular filter samples. This includes damaged or smudged areas on the filter surface, lack of uniform sample deposition, unattached particulate or debris, unusually heavy-appearing deposit concentration, or other evidence of unusual condition.

Mount any damaged areas removed prior to sample preparation on glass slides using double-sided adhesive and carefully measure the diameter of the effective filter area. The total effective filter area and damaged areas of sample removed should be accurately recorded for subsequent calculation of asbestos concentrations.

### C.2 Sample Preparation

#### C.2.1 Samples on Millipore Filters

In the original sample dish, cut a 90° radial section of the original 47-mm filter sample with a clean, single-edged razor blade. Transfer the quarter section with stainless steel forceps to a clean 1 in. x 3 in. glass slide, and cut again into smaller wedges to fit into the glass ashing tube (approximately 15-mm long). Transfer the wedges by forceps to clean, numbered ashing tubes. Place the tubes in an LFE 504 low temperature plasma oven, one sample tube and one laboratory control tube per ashing chamber. The laboratory control tube may either contain a blank Millipore filter or be run as an empty tube. Maintain the ashing process at 450 watts for 2 hr.

Upon removal from the oven, treat the ashing tubes as follows. Place the tube in an ultrasonification bath. Pour 1 to 2 ml of 0.22  $\mu\text{m}$  filtered Millipore-Q water into the tube from a clean 100 ml graduated cylinder. Sonicate (at 40 milliamperes) the sample vigorously for approximately 5 min and transfer it to a clean 150 ml glass beaker. Rinse the tube by additional ultrasonification two or three times more using a few milliliters of filtered water each time, and transfer the contents to a

150 ml sample beaker. Add the remaining volume (up to 100 ml) of filtered water and sonicate again the entire suspended sample or blank, so that the total time of dispersion in the sonicator takes at least 20 min. Use a clean glass rod to stir the suspended sample while it is being sonicated.

Divide the 100 ml fraction into three aliquots: 10, 20, and 70 ml, prepared in that order. Using a 25-mm Millipore filter apparatus, place a 0.2  $\mu$ m Nucleopore polycarbonate filter on top of an 8.0  $\mu$ m mixed cellulose ester Millipore backup filter. Wet the filters by aspirating approximately 10 ml of filtered deionized water. Stop aspiration, pour in the first sample aliquot or portion thereof, and begin the aspiration procedure again. Carefully add the remaining sample volume without disturbing the flow across the Nucleopore filter surface. The suspended sample may be resonicated or stirred between filtration of the aliquots.

When the sample is deposited, carefully transfer the Nucleopore filter to a clean, labeled (sample number, date, and aliquot size) 1 x 3 in glass slide. Discard the Millipore backup filter.

When dry, attach the 0.2  $\mu$ m Nucleopore filter tautly to the slide with transparent tape. Coat the filter with an approximately 40-nm-thick carbon film (National Spectroscopic Laboratories carbon rods) by vacuum evaporation. The film thickness need be sufficient only to provide support for the deposit sample.

Transfer the polycarbonate filter deposit to a 200-mesh electron microscope copper grid (E. F. Fullam) by first cutting a 3-mm-square portion from the filter using a clean, single-edged razor blade. Place this deposit side down on the electron microscope (EM) grid which, in turn, has been set upon a small, correspondingly labeled portion of lens tissue paper. Place the film, grid, and lens paper on a Jaffe dish consisting of a copper screen supported on a bent glass rod in a covered 90-mm glass petri dish. Pour reagent grade chloroform (J. T. Baker Company) into the dish to saturate the lens paper without submersing the grid and sample. Keep the dish covered at room temperature for 2 hr. Shift the prepared sample to a clean petri dish with fresh chloroform. Heat to 40°C for 10 min to provide a washing procedure.

While it is still wet, place the sample grid in a small gelatin capsule. Tape the capsule to the slide that has the remaining coated polycarbonate filter, and store until analysis.



### C.2.2 Samples on Nucleopore Filters

The above ashing and refiltering procedures are unnecessary for samples collected directly on nucleopore filters. Instead, the filter is carbon-coated and transferred to an EM grid as described in the preceding three paragraphs.

### C.3 Microscopic Procedures

Select a sample or, for samples ashed and refiltered, start with the 70-ml aliquot of filtered material. Examine the EM grid under low magnification in the transmission electron microscope to determine its suitability examination under high magnification. Ascertain that the loading is suitable and is uniform, that a high number of grid openings have their carbon film intact, and that the sample is not contaminated excessively with extraneous debris or bacteria.

Scan the EM grid at a screen magnification of 20,000X. Record the length and breadth of all fibers that have an aspect ratio of greater than 3:1 and have substantially parallel sides. Observe the morphology of each fiber through the 10X binoculars and note whether a tubular structure characteristic of chrysotile asbestos is present. Switch into selective area electron diffraction (SAED) mode and observe the diffraction pattern. Note whether the pattern is typical of chrysotile or amphibole, ambiguous, or neither chrysotile nor amphibole. Use energy dispersive X-ray analysis where necessary to further characterize the fiber. Take pictures as desired representing the sample type, fiber/particulate distribution, or characteristic SAED patterns of chrysotile and specific amphibole types.

Count the fibers in the grid openings until at least 100 fibers, or the fibers in a minimum of 10 grid openings, have been counted. Once counting of fibers in a grid opening has started, the count shall be continued though the total count of fibers may be greater than 100.

To ensure uniformity of grid opening dimensions, examine several 200-mesh grids by optical microscopy and measure roughly 10 openings per grid. Average these dimensions to provide a standard grid opening area.

#### C.4 Calculations

Calculate from the following equation fiber number concentration expressed as the total number of fibers/volume of air:

$$\text{Fiber counts (f/m}^3\text{)} = \left( \frac{\text{number of fibers counted}}{\text{number of grids examined}} \right) \left( \frac{\text{total effective filter area, cm}^2}{\text{average area of an EM grid opening, cm}^2} \right) \left( \frac{\text{dilution factors}^*}{\text{volume sampled, m}^3} \right)$$

Calculate fiber mass for each type of asbestos in the sample by assuming that the breadth measurement is a diameter; thus, the mass can be calculated from:

$$\text{Mass } (\mu\text{g}) = \frac{\pi}{4} \cdot (\text{length, } \mu\text{m}) \cdot (\text{diameter, } \mu\text{m})^2 \cdot (\text{density, g/cm}^3) \cdot 10^{-6}$$

The density of chrysotile is assumed to be 2.6 g/cm<sup>3</sup>, and of amphibole, 3.0 g/cm<sup>3</sup>. The mass concentration for each type of asbestos is then calculated from:

$$\begin{array}{l} \text{Mass Concentration} \\ (\mu\text{g/m}^3) \text{ of a} \\ \text{Particular Type} \end{array} = \frac{\text{Total Mass of All} \\ \text{Fibers of that Type } (\mu\text{g})}{\text{Volume of Air Sampled (m}^3\text{)}}$$

Record the fiber bundles and clusters as such, but do not include them in the mass calculation or the fiber count. The fiber clusters and fiber bundles are not included in the mass calculation because (1) it is difficult to assign the third dimension to the two-dimensional observation of the aggregates, (2) it is difficult to determine void space within bundles and clusters, and (3) since the bundles and clusters make up only about 2% of the item count, one cannot be certain of the even distribution throughout the filter.

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\* Dilution factors take into account sample dilution during ashing and refiltering and transfer to the EM grid.

Appendix D. Quality Assurance Plan

In Preparation